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THE MICROSTRUCTURAL AND PROPERTY CHANGES  
OF LASER TREATED ELECTRODEPOSITS

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January 1981



US ARMY ARMAMENT RESEARCH AND DEVELOPMENT COMMAND  
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## INTRODUCTION

For the surface protection of high strength steels against severe high temperature gases, attention has often been directed towards the use of refractory metal coatings. Electrodeposition would be an economical way for depositing refractory metals but is limited to rhenium<sup>1-3</sup> which is a brittle and costly metal. Amongst the successful metals that can be electrodeposited is high contractile (HC) or hard chromium. This has shown the ability to withstand severe wear and high temperature environments. In spite of its success however, chromium is also brittle and possesses an inherent crack pattern which leads to excessive spalling or flaking when subjected to high stresses. Evidence of the latter can be seen in Figure 1.

Low contractile (LC) chromium<sup>4-6</sup> is a more ductile coating and is less susceptible to spalling. It, however, lacks some of the mechanical properties that one requires under high loads.

Attempts to improve the properties of electrodeposited chromium or refractory metals by alloying with iron group metals have also fell far short of the ideal coating. In such cases, the deposits were found to be brittle, and non-adherent to steel substrates.<sup>7-10</sup>

Heating chromium plated high strength structural steels to an elevated temperature for diffusing the chromium with the substrate to produce suitable alloying at the surface has also been attempted. The major disadvantages of this approach are the formation of brittle diffusion layers,<sup>11</sup> the decrease in the mechanical properties of the bulk structure, and the distortion which may occur.

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References are listed at the end of this report.

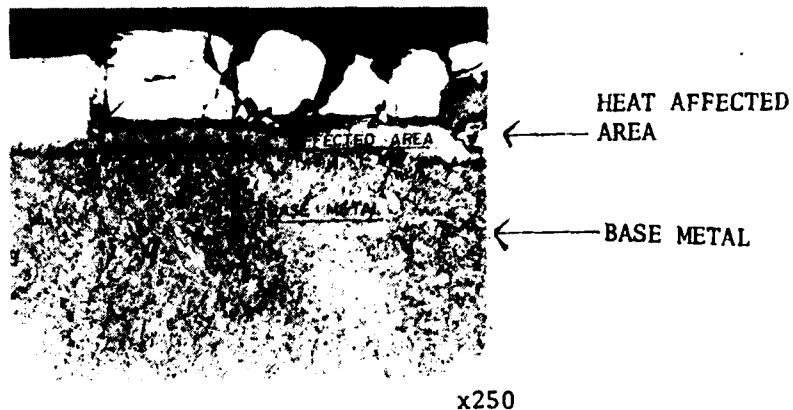


Figure 1. Cross-sectioned chromium plated steel segment taken from the bore of a gun tube which fired hypervelocity rounds.

In view of the above, recent attention has been given to a new approach for altering the properties of a metal deposit, which involves the heat treatment of the plated surface using LASERS. This present investigation is basically a preliminary study which advances the efforts of a previous investigation on the behavior of LASER treated chromium deposits on steel substrates.<sup>12</sup>

During the past two decades the LASER (Light Amplification by Stimulated Emission of Radiation) has advanced from a laboratory curiosity to a precious tool promoting numerous advances in metal processes. The LASER has attracted the metallurgists, primarily because it can focus vast amounts of energy very rapidly and with pinpoint accuracy upon a metal surface.

The introduction of the LASER in the 1950's and the development of high power gas LASERS in the 1960-70's have led to rapid advancement in industrial applications for the processing of metals. Due to these advances, LASERS are



being successfully applied in the field of cutting, drilling, welding, and surface heat treating of metals and alloys.

Solid LASERS were the first to be employed but are limited since they can function in a pulsed rather than continuous mode and due to their melting point could absorb only a limited power input. In 1965, the concept of the gas LASERS was introduced whereby high energy could be radiated in the continuous mode as well as in pulses. Because of the high power capability, the gas LASER was employed for this present study.

A LASER technique<sup>13</sup> developed in recent years for the thermal treatment of a metal surface is called Laserglazing\*. Such a technique involves the melting of thin surface layers (e.g., steels) at a high melting efficiency, resulting in rapid solidification and cooling to achieve a variety of microstructures and properties without affecting the bulk substrate. This is accomplished by rapidly traversing the surface of a material with a LASER beam focused to a power density range of  $10^4 - 10^7$  W/cm<sup>2</sup>.

In order to compare Laserglazing with other material processes using LASERS, a spectrum for operational boundaries is shown in Figure 2 in which LASER power densities are related to interaction time.

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\*LASERGLAZE - Trade Mark by United Technologies Corporation, East Hartford, CT.

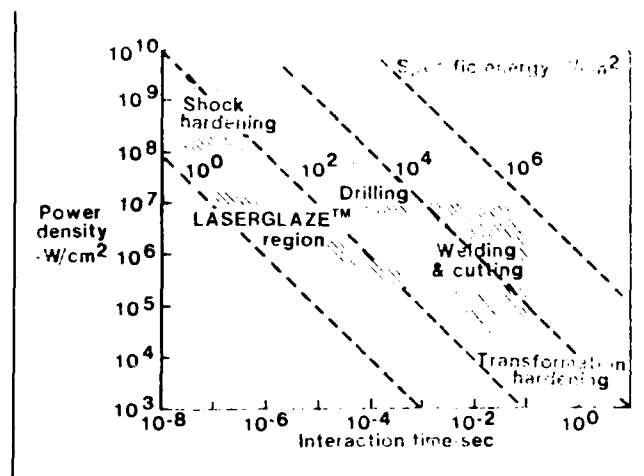


Figure 2. Operational boundaries for laser beam-material interaction for various processes.<sup>13</sup> The diagonals are lines of constant specific energy with the values shown.

The significant difference which results in the structure of a Laser-glazed surface can be clearly realized when comparison is made with the structure obtained in conventional casting of an alloy. In the casting of a bulk alloy, the phases with high melting points are difficult to melt and phase separations occur. Such structures provide non-uniform properties. In the case of a Laserglazed surface, the melted layer is thin and since the substrate remains cold, rapid solidification occurs thus reducing those problems associated with slow cooling.<sup>13</sup>

This technique of achieving homogeneous phase transformations in the surface with sufficient control of surface depth has formed the basis of this

present investigation. Also the ease of automating the process for a variety of geometrical shapes offers a number of economic advantages.

The work of Montgomery<sup>12</sup> appears to be the first study dealing with the Laser treatment of electrodeposited chromium to improve properties and adhesion. This work involved LASER heating of the plated surface with transformation of the steel substrate approaching depths .203-.254  $\mu\text{m}$  (80-100 mils). The coating thicknesses ranged from 25-317  $\mu\text{m}$  (1-12.5 mils) and the speeds of LASER beam travel (which controls interaction time) were 25-102 cm/min (10-40 in./min). Results of the above study revealed suitable Fe-Cr alloying but some undesirable embrittlement of the softened unalloyed portions of the deposit and indications of the formation of intermetallic compounds of Cr-Fe carbide. The latter deposits resulted in premature failure in wear tests.

#### OBJECTIVE

In this present investigation, the effects of intermediate layers of LC chromium and cobalt are investigated as a function of exposure time as a means of optimizing structural and interfacial characteristics.

In view of the possibility of the formation of brittle Cr-Fe carbide phases at the interface and the potential erosion resistance properties of cobalt, the steel/Co/Cr combination was included into the present study to compare its behavior with the steel/Cr combination.

Low contraction (LC) chromium has been introduced in this study because it is known to have less cracks, contract less and subsequently have less oxide<sup>4-6</sup> than HC chromium, and therefore would be expected to behave differently during LASER treatment.

## EXPERIMENTAL PROCEDURE

### Electrodeposition

All test coatings were plated on flat steel strips (cold rolled SAE 4340\*) which measured approximately 1.9 cm wide, 15 cm long, and 0.16 cm thick (0.75 x 6 x 1/16 in.). The test panels were first cleaned by soaking in a hot caustic solution and scrubbing, followed by a hot water rinse. After rinse the panels were electropolished to remove 25-50  $\mu\text{m}$  (1-2 mils) of the surface. Without delay the panels were immediately rinsed and plated on one side to produce deposits of:

- (a) 84.4  $\mu\text{m}$  (3.4 mils) of Cr on steel (42.4  $\mu\text{m}$  of HC chromium over 42.2  $\mu\text{m}$  of LC chromium).
- (b) 84.4  $\mu\text{m}$  (3.4 mils) of Cr on 95.3  $\mu\text{m}$  (3.75 mils) of Co on steel.

#### Bath Formula and Process Controls:

1.

#### Cobalt Plating

Cobalt Sulfamate . . . . .	320 g/l (43 oz/gal)
Boric Acid . . . . .	22-30 g/l (3-4 oz/gal)
Cobalt Bromide Concentrate . . . . .	45-67 g/l (6-9 oz/gal)
Current Densities. . . . .	11 amp/dm <sup>2</sup> (100 amp/ft <sup>2</sup> )
Temperature. . . . .	49 $\pm$ 1C (120 $\pm$ 2°F)
pH . . . . .	4.0 $\pm$ .2
Sp gr. . . . .	1.25 - 1.28 (29-31 Baume')
Anodes (bagged). . . . .	Cast Cobalt

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\*No concern was given to the tempering of the test coupons prior to plating, in view of the altering of the underlying steel during LASER heating and quenching.

2.

### Chromium Plating

CrO <sub>3</sub> . . . . .	250 g/l (33 oz/gal)
H <sub>2</sub> SO <sub>4</sub> . . . . .	2.5 g/l (.33 oz/gal)
Sp gr. . . . .	1.17 - 1.18 (21-22 Baume')
Current density. . . . .	32 amp/dm <sup>2</sup> (300 amp/ft <sup>2</sup> ) For HC chromium; 64 amp/dm <sup>2</sup> (600 amp/ft <sup>2</sup> ) For LC chromium
Temperature. . . . .	54°C ± 1°C (130°F) HC chromium; 85°C ± 2°C (185°F) LC chromium
Anodes . . . . .	Lead-Tin

3.

### Electropolishing

H <sub>2</sub> SO <sub>4</sub> . . . . .	50% (vol.)
H <sub>3</sub> PO <sub>4</sub> . . . . .	50% (vol.)
Current density. . . . .	32 amp/dm <sup>2</sup> (300 amp/ft <sup>2</sup> )
Temperature. . . . .	43°C ± 1°C (110°F)

### Laser Treatment

A high-power electric discharge cross beam CO<sub>2</sub> LASER, operated as an unstable resonator (with a constant power setting of 6 KW), was employed in a continuous mode to heat the plated specimens for the present study with the following parameters:

Total energy applied to specimen surface . .	4.7 KW max
Power density. . . . .	2.3 x 10 <sup>6</sup> W/cm
Wavelength . . . . .	10.6 μm
Focal length of beam . . . . .	47.6 cm (18 3/4 in.)
Diameter of focal spot . . . . .	.051 cm (20 mils)

Scanning speed (linear). . . . . 5.1-95.5 cm/sec  
 Shielding (100% He). . . . . 2.8 m<sup>3</sup>/hr  
 Focusing Mirror. . . . . Copper, off-axis parabola

The LASER treatment was carried out by mounting the plated specimens on a large aluminum rotating horizontal disk so that the LASER beam (mounted in a stationary position) would make contact with the surface along the length of the specimen during various preset speeds. The speed of the rotating disk and the number of revolutions (or passes) controlled the interaction time (i.e., time specimen was exposed to the LASER beam). In order to prevent oxidation of the plated surfaces during the LASER treatment, a helium environment was utilized.

Nominal exposure times, calculated from specimen speeds were 0.5, 10, and 50 ms, resulting in the surface patterns shown in Figure 3. The widths of the affected trails were increased by displacing the specimens a small distance laterally from the fixed focal point of the LASER beam before each pass.

#### Specimen Evaluation

Examination of the samples consisted of metallographic, microhardness, and microprobe analysis.

#### Photomicrographs

Specimens were cross-sectioned normal to the LASER treated paths and mounted and polished using diamond abrasives. The polished specimens were etched as follows: the steel etchant used consisted of two grams picric acid 100 ml ethyl alcohol plus seven drops concentrated hydrochloric acid. The chromium and cobalt etchant used consisted of a solution of 60 parts lactic acid, 30 parts HNO<sub>3</sub> + 5 parts HF, by swabbing 10-15 sec. The photomicrographs were made with a Polaroid camera using a Leitz MM5 Research Metallograph.

### Hardness Measurements

Hardness measurements were made on the cross sections of the deposited specimens with a Wilson Tukon microhardness tester using a 50 gm load and are reported on the Knoop scale (KHN).

### Microprobe Analysis

Coatings were analyzed with the scanning electron microprobe (Materials Analysis Corporation Model 4005) using a specimen current to approximately 10-20 nA and 25 KV. The specimens were scanned along the two directions of the mounted cross sections (i.e., from the substrate to the top layer of the deposit and along the center of the coating parallel to the substrate).



x3

Figure 3. Surface view of laser treated chromium plated specimen showing the altered trails caused by the laser beam during specimen travel.

## RESULTS AND DISCUSSION

### As-Plated Cobalt/Chromium on Steel

A cross-section of the as-plated duplex structure of electrodeposited chromium over cobalt on steel is shown in Figure 4. The etched layers of chromium reveal the coarser grained columnar structure of the LC versus the HC deposit (see top inset). The microstructure of the cobalt is also typically columnar and shown in the lower inset. The steel is shown to be a spheroidal structure.

The hardness and characteristics of chromium deposits directly onto steel substrates were found to be approximately the same as those with the cobalt underlay.

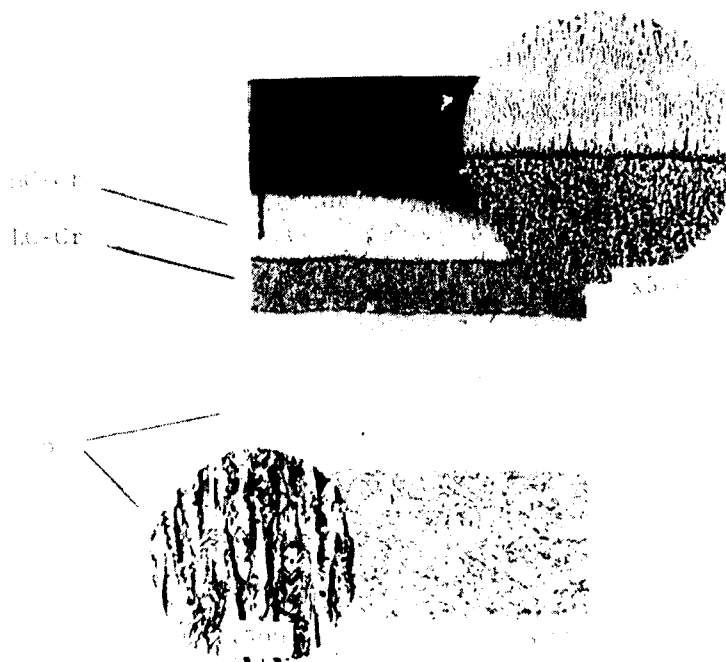


Figure 4. Cross-section of an as-plated duplex structure of electrodeposited chromium over cobalt onto steel.



### LASER Treated Cr on Steel and Co/Cr on Steel

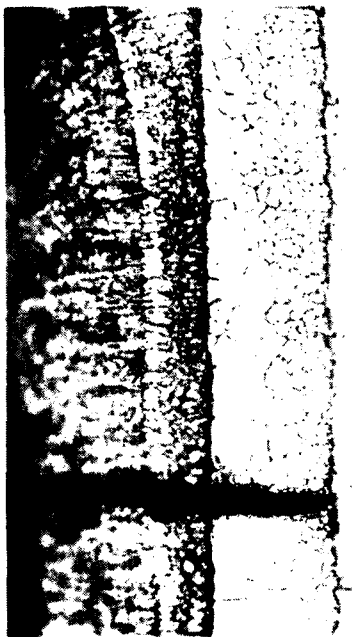
A. Exposure Time of 0.500 ms - This test run was accomplished by a single pass across the LASER beam with a specimen travel speed of 95.5 cm/sec (188 ft/min).

Chromium on Steel - The LASER beam affect on the microstructure of this trial run is shown in Figure 5(a)\*. As can be seen, the microstructure of the LC chromium is no longer columnar but has been transformed (through recrystallization and grain growth) to a somewhat equiaxed structure with the grains progressively coarser towards the edge of the LASER path. The HC chromium top layer which obviously experienced higher temperatures, still reveals some columnar structure, but has significantly coarsened. The underlying steel substrate reveals some early signs of phase reaction. The substrate exhibits an increase and the chromium a decrease in hardness as shown in Figures 6 and 7.

Cobalt/Chromium on Steel - The microstructural changes of the chromium layers over the cobalt are approximately the same as the chromium/steel specimens (Figure 5(b)) with a corresponding decrease in hardness. An interesting observation is the widening of the initial cracks in the chromium. The cobalt and underlying steel revealed no changes in microstructure although the hardness profiles (Figures 6 and 7) revealed a slight hardness decrease for cobalt and slight increase in the steel just beneath the cobalt.

B. Exposure Times Of 10 ms - This LASER treatment was performed with a specimen linear speed of 25.9 cm/sec (51 ft/min) and the specimen passing across the LASER beam five times for a total interaction time of 10 ms.

\*The diamond shaped indentations shown in the photomicrographs are due to the hardness measurements taken.



(a) Cr on Steel  
x500



(b) Co/Cr on Steel  
x500

Figure 5. Cross-section of heat affected electrodeposited steel specimens after laser inter-  
action times of 0.500 ms. Note the recrystallized grain structure of the chromium  
layers.

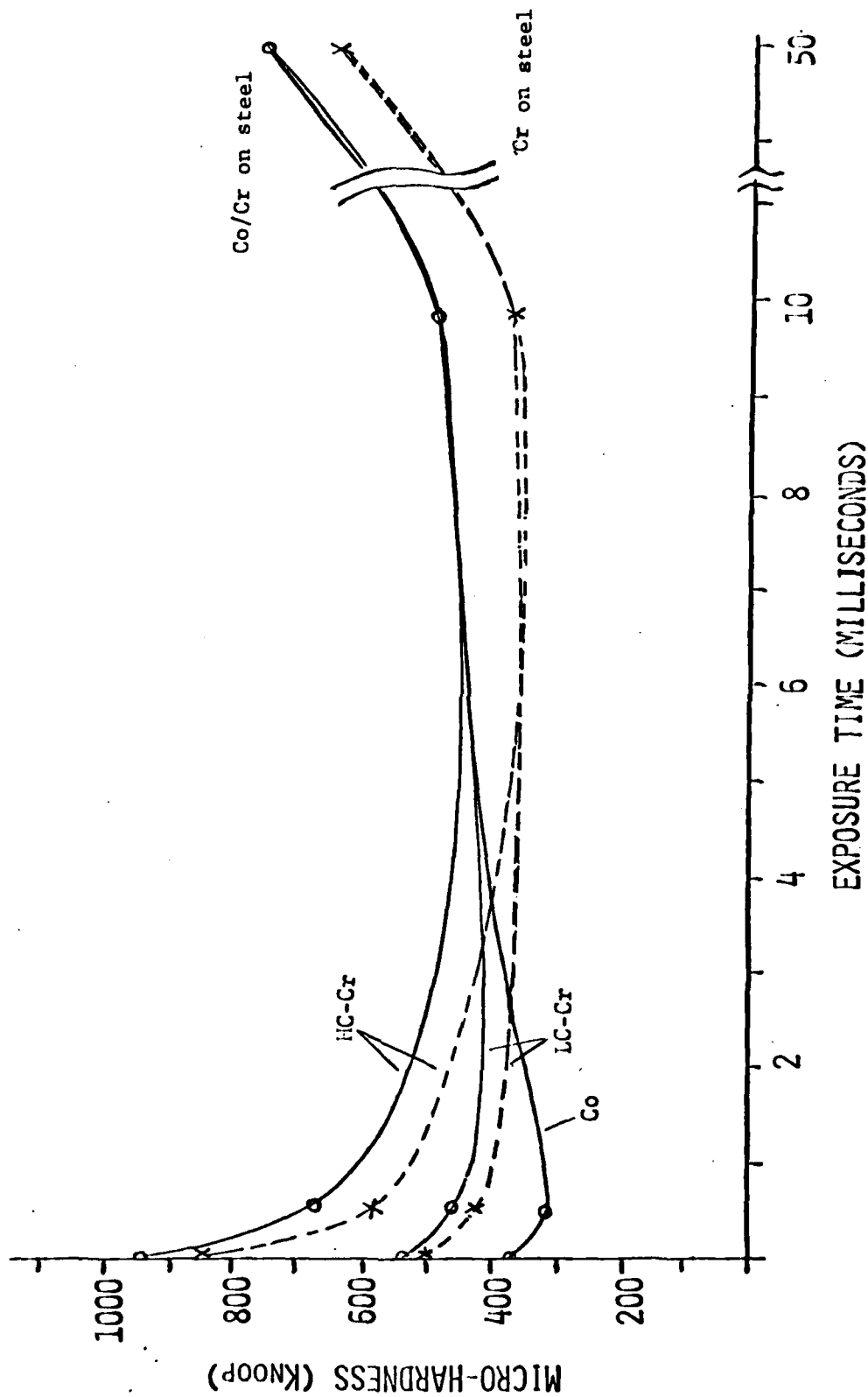


Figure 6. Relation of hardness to interaction time for chromium and cobalt coatings.

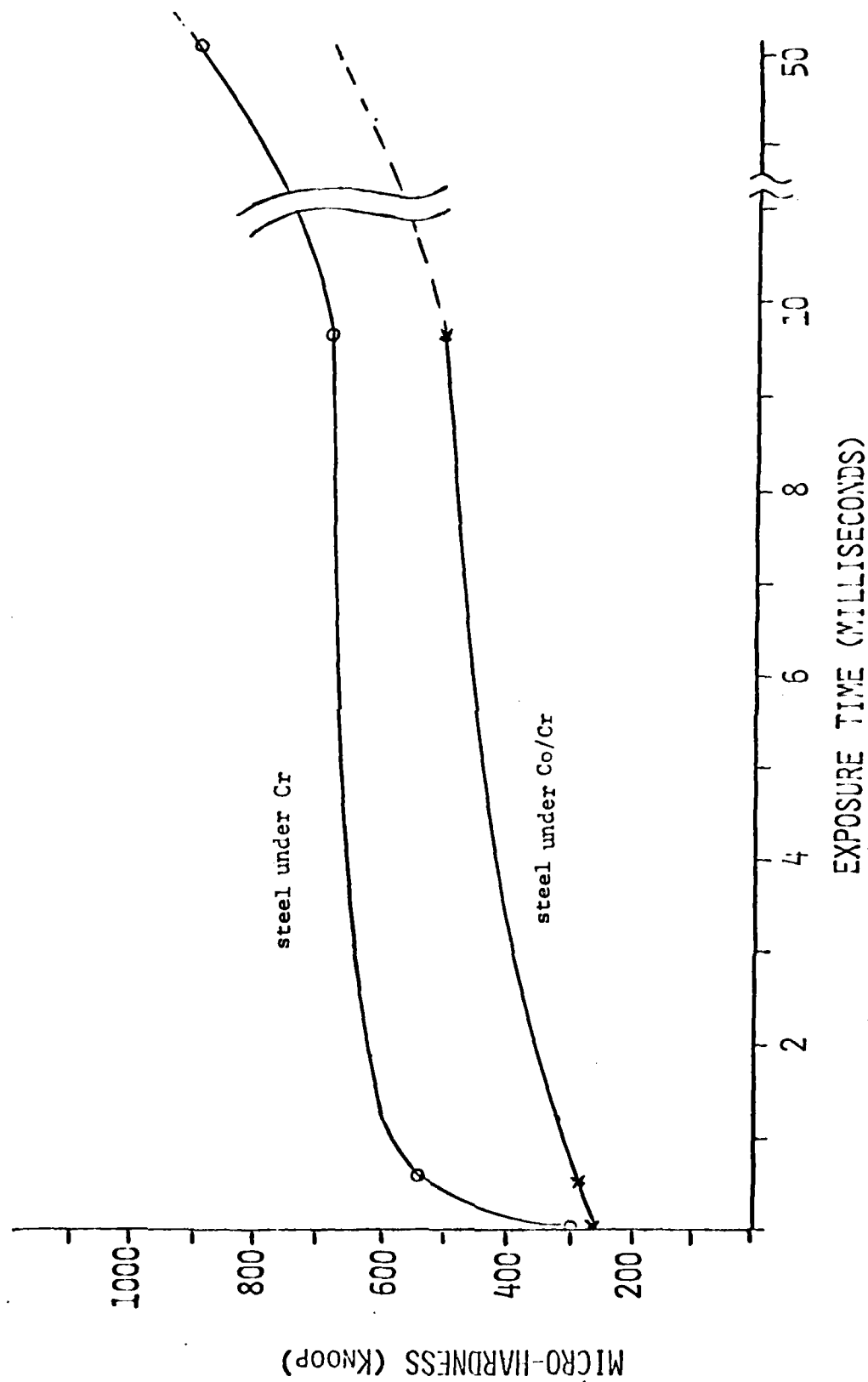


Figure 7. Relation of hardness to interaction time of the underlying steel substrate (HAZ).

The specimen was moved .025 cm (10 mils) laterally prior to each overlapping pass in order to widen the heat affected zone. The total lateral displacement for the four succeeding passes was approximately .152 cm (60 mils).

Chromium on Steel - Figure 8(a) shows extensive changes in microstructure of the chromium and underlying steel with no visibility of the HC-LC interface. In addition, the coating did not respond to microetching indicating changes in chemical resistance. Microprobe analysis has shown indications of varying concentrations of iron in the chromium. One can see a heat affected zone (HAZ) in the underlying steel with significant nonhomogeneity at the steel/Cr interface. Another significant observation is the existence of internal cracks in the chromium coatings (possibly related to the high quenching rates or shrinkage during cooling). Figure 8(b) is a cross-section of an area adjacent to the LASER beam path which reveals the thermal alterations due to edge affects. Material from the substrate appears to be partially drawn up through the cracks in the chromium. This indicates that the substrate could possibly approach the melting temperature at the bottom of pre-existing cracks. The entry of material into the cracks was observed to a greater extent in the previous study.<sup>12</sup>

Cobalt/Chromium on Steel - Figure 9 clearly depicts the significant phase changes in the steel/Co/Cr system for the same interaction time (10 ms). The extreme left of the view (a) represents the center of the narrow LASER beam path where the coatings appear to have transformed into a single and homogeneous layer which again did not respond to etching. However, the section on the right side of the photomicrograph (i.e., less heat input) clearly reveals the chromium and cobalt layers. The chromium is shown to be



x500

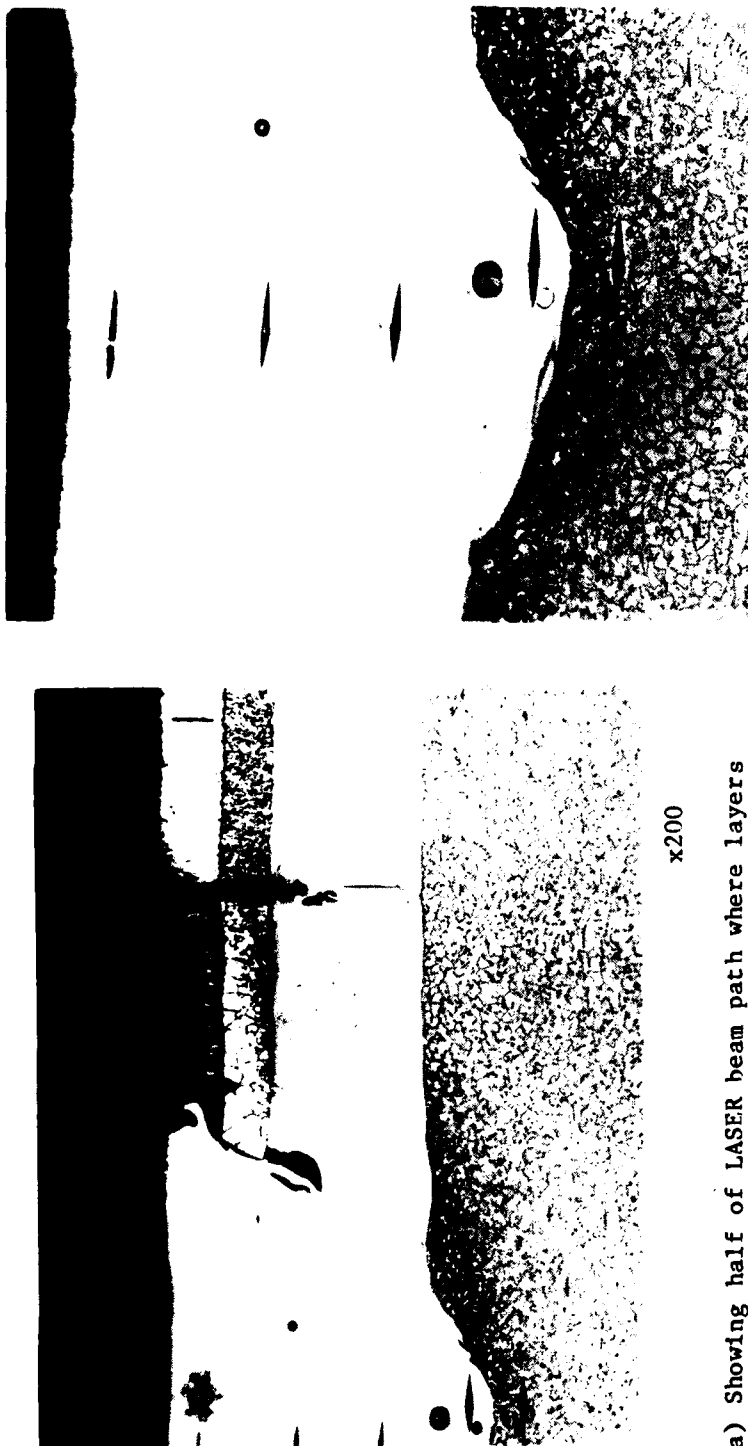
(b) Edge of LASER beam path where layers are still distinguishable due to partial heating. Note the nonhomogeneity of the thermally altered underlying steel.



x300

(a) Transformed Cr-Fe Coating which lies in the center of LASER beam path. Note internal cracks.

Figure 8. Cross-section of heat affected electrodeposited Cr on Steel specimens after LASER interaction times of 10 ms.



(a) Showing half of LASER beam path where layers are transformed into a single coating. Note the edge affects due to partial heating where layers are distinguishable.

x200

(b) Showing center view of the transformed coating path. Note HAZ of the underlying steel.

x300

Figure 9. Cross-section of heat affected electrodeposited steel/Co/Cr specimen after LASER interaction time of 10 ms.

progressively recrystallized as it approaches the beam path. The interface between these two zones reveals a number of voids and disarranged structure due to the end effects of the LASER beam where temperature gradients are extremely high.

Figure 9(b) shows the overall view of the transformed coating which corresponds to the center of the LASER beam path.

Examination of the underlying steel reveals a transformation which appears to be untempered martensite. These microstructural changes of the coatings and substrate, again are shown to correlate with the corresponding hardness changes in Figures 6 and 7.

C. Exposure Time of 50 ms - This LASER treatment involved a specimen linear speed of 5.1 cm/sec (10 ft/min). Again, the specimens were passed across the LASER beam five times for a total interaction time of 50 ms. The specimen lateral displacements for these trials were .051 cm (20 mils) for each succeeding pass for a total displacement of .254 cm (100 mils).

Chromium on Steel - The results from this LASER treatment shown in Figure 10 reveals phase transformations which extend through the complete thickness of the specimen. Again, neither the affected coatings or the steel responded to microetching confirming that significant phase changes have taken place. The structural changes as a result of this treatment was accompanied with an increase in microhardness (as shown in Figure 6). This could be due to solid solution strengthening of the Cr-Fe formation which was identified by microprobe analysis. A hardness plot extending through the full thickness of .157 cm (62 mils) of the specimen reveals a relatively uniform level as shown in Figure 11.



Figure 10. Cross-section of heat affected electrodeposited Cr on steel specimen after LASER interaction time of 50 ms. Phase transformations have advanced through the complete thickness of the specimen.



x50

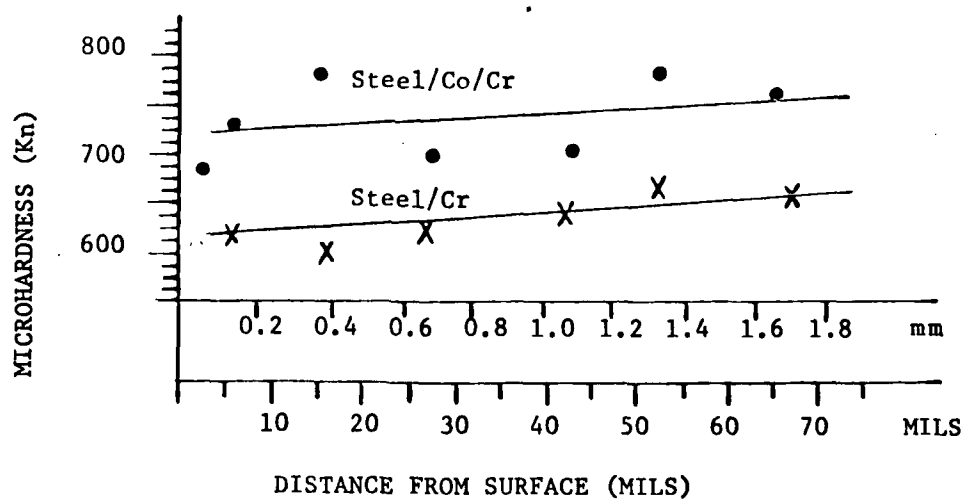


Figure 11. Hardness plot through specimen thickness for the interaction time of 50 ms.

Cobalt/Chromium on Steel - The 50 ms LASER treatment of the steel/Co/Cr system appears to have experienced the same extensive phase transformations and the metallographic view appeared identical to the Cr on steel as shown in Figure 10. The change, was again, accompanied by an increase in hardness which was comparatively greater than the Cr on steel specimens (see Figures 6 and 11).

#### CONCLUSIONS

(a) The LASER technique employed was shown to affectively confine the heating of the surface to controllable depths. At an exposure time of .5 ms, grain growth in the chromium occurred with a decrease in hardness and negligible interfacial effects. At an exposure time of 10 ms, total recrystallization of the chromium occurred. In the cobalt-chromium duplex coating, complete alloying occurred. At 50 ms, alloying through the total .157 cm (62 mils) specimen thickness occurred.

(b) The introduction of cobalt as an underlay for chromium was found to be beneficial in eliminating phase reactions between steel and chromium. Also the steel/Co/Cr system exhibited less cracking and porosity and possessed greater hardness after treatment than the Cr on steel system.

(c) The LC chromium layers exhibited less cracking and porosity than the HC chromium following the 0.5 ms and 10 ms LASER treatments. After the 50 ms treatment, no differences could be observed.

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